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(51) INT CL³
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U1S 1513 2323 B1B

(56) Documents cited
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(58) Field of search
B1B

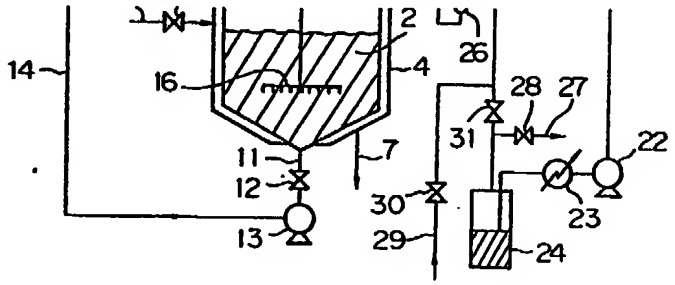
ERRATUM

SPECIFICATION NO 2142549A

Front page Heading (57) Abstract Line No 14 after an for insert read inert
Page No 2 Line No 86 after and for boow read blow

THE PATENT OFFICE
13 January 1986

the inert gas containing the low-boiling-point fraction, an insert gas circulating mechanism arranged to connect the inert gas withdrawal mechanism, and a low-boiling-point fraction recovery mechanism arranged in the circulating mechanism to cool and condense the low-boiling-point fraction.



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(54) Evaporation and distillation apparatus utilizing inert gas

(57) An evaporation and distillation apparatus for recovering a low-boiling-point fraction of distillate from a liquid containing a plurality of components comprises a heating mechanism to heat the liquid, a blowing mechanism for blowing an inert gas into the liquid and evaporating a low-boiling-point fraction together with the inert gas, an inert gas withdrawal mechanism arranged in the upper portion of the apparatus to withdraw the inert gas containing the low-boiling-point fraction, an insert gas circulating mechanism arranged to connect the inert gas withdrawal mechanism, and a low-boiling-point fraction recovery mechanism arranged in the circulating mechanism to cool and condense the low-boiling-point fraction.

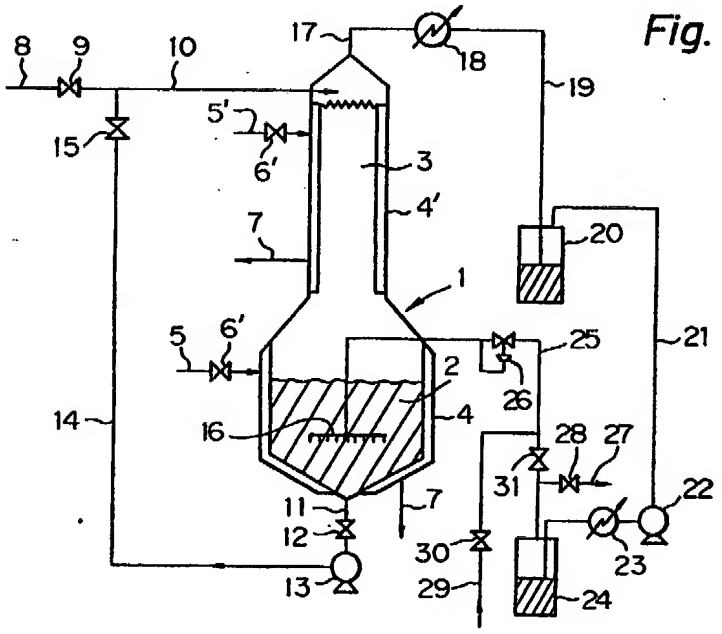

Fig. 1

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Fig. 1

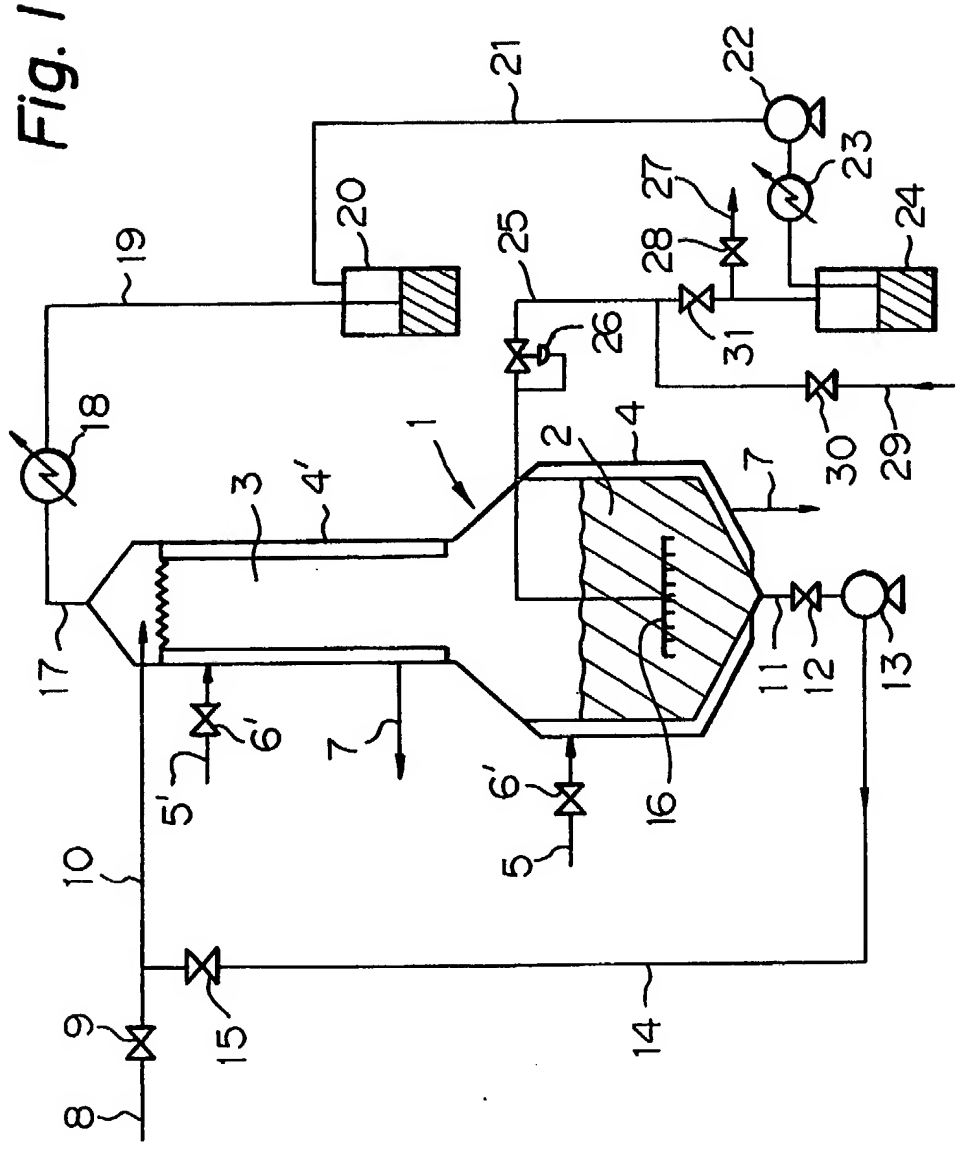
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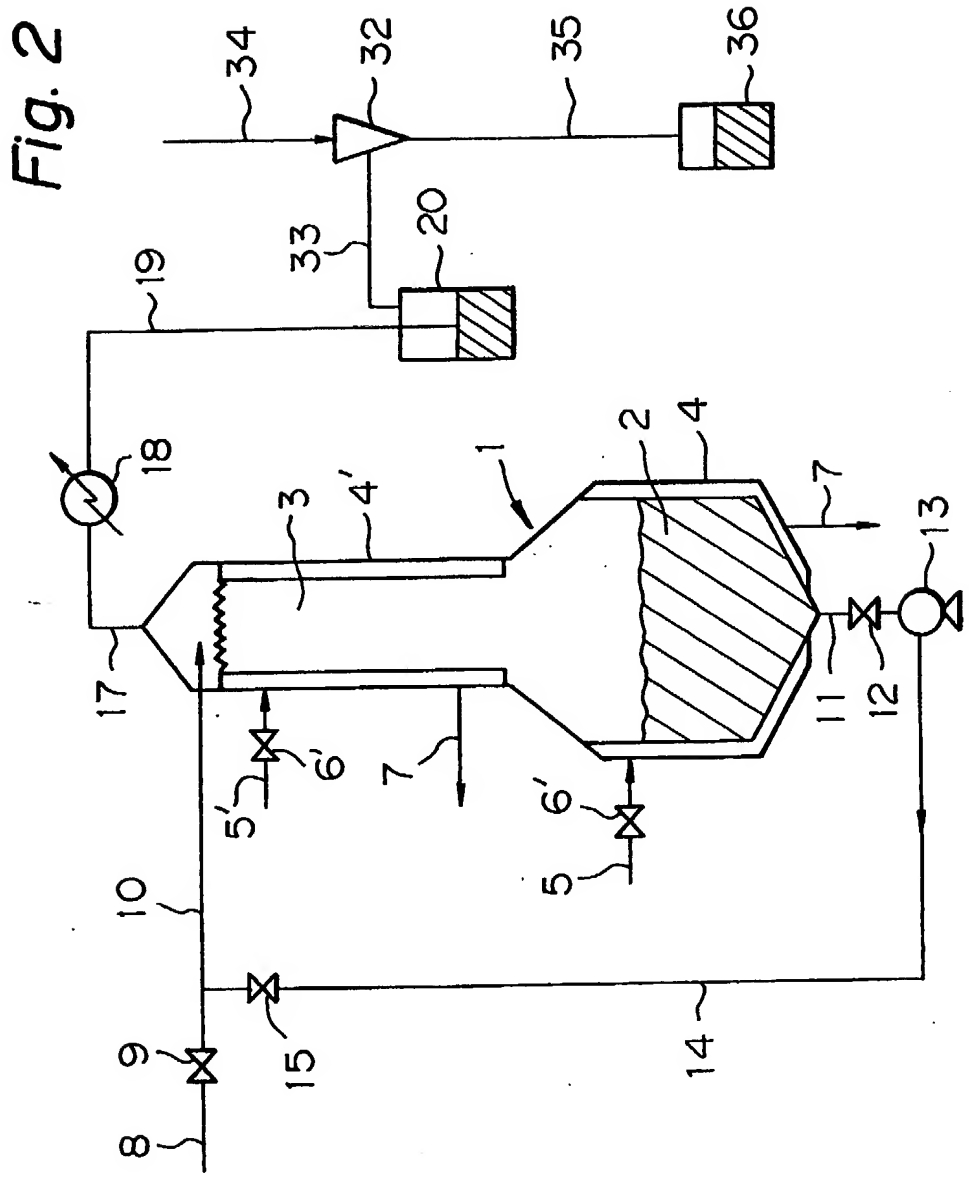
Fig. 2



SPECIFICATION

Evaporation and distillation apparatus utilizing inert gas

Background of the invention

(1) Field of the invention:

The present invention relates to an evaporation and distillation apparatus utilizing an inert gas. More particularly, the present invention relates to an apparatus for recovering a low-boiling-poing fraction of distillate from a liquid containing a plurality of components by evaporation and distillation at a high efficiency in a closed system.

(2) Description of the prior art:

When a high-boiling-point fraction is to be recovered by the evaporation or distilled process, the boiling point of the fraction used to be reduced under high vacuum and/or blowing steam into the liquid.

In the case where the evaporation or distillation is carried out under a high vacuum, usually a water jet or steam jet is used instead of a high vacuum oil pump. In this case, a large quantity of water or steam shall be consumed and some parks of the distillate is discarded in water steam drain or water.

Moreover, in case of the steam distillation or evaporation, a large quantity of steam is cooled and condensed together with the fraction of distillate in a condenser, and it becomes necessary to remove water from the fraction of distillate. Of course, also the bottom residue should naturally contain water.

In addition, in the case where the liquid to be treated is, for example, a mixture of an oil or fat and a solvent, since there is an affinity between the two components, even if the above-mentioned means is adopted, it is generally difficult to perform separation of the solvent completely.

Summary of the invention

It is therefore a primary object of the present invention to provide an evaporation and distillation apparatus in which a low-boiling-point fraction of distillate is separated and recovered from a liquid containing a plurality of components at a high efficiency by utilizing an inert gas.

Another object of the present invention is to provide an evaporation and distillation apparatus in which a desired fraction of distillate can be recovered with higher yield and less heat requirement and the residual liquid contains for less distillate than by other conventional method without causing thermal deterioration or oxidation of components of a liquid to be treated.

Still another object of the present invention is to provide an evaporation and distillation apparatus in which the above-mentioned evaporation or distillation is carried out in a closed system (closed circuit) to prevent escape of distillate to the outside.

More specifically, in accordance with the present invention, there is provided an evaporation and distillation apparatus for recovering a low-boiling-point fraction of distillate from a liquid containing a plurality of components, said apparatus comprising a heating mechanism to heat the liquid, a blowing mechanism for blowing an inert gas into the liquid and evaporating a low-boiling-point fraction together with the inert gas, an inert gas withdrawal mechanism arranged in the upper portion of the apparatus to withdraw the inert gas containing the low-boiling-point fraction, an inert gas circulating mechanism arranged to connect the inert gas withdrawal mechanism with the blowing mechanism, and a low-boiling-point fraction recovery mechanism arranged in the circulating mechanism to cool and condense the low-boiling-point fraction.

Brief description of the drawings

Figure 1 is a view illustrating arrangement of an embodiment of one example of the present invention.

Figure 2 is a view illustrating arrangement of a conventional apparatus.

Detailed description of the preferred embodiments

According to the present invention, an inert gas such as nitrogen or carbon dioxide gas is used instead of steam in the steam distillation operation, and this inert gas is introduced into a liquid to reduce the partial vapor pressure of the distillate, whereby the boiling point of the liquid is reduced and a low-boiling-point component in the liquid is evaporated and separated together with the inert gas from the liquid.

The inert gas mixture containing the evaporated fraction is withdrawn under atmospheric or reduced pressure from the evaporation or distillation apparatus and the mixture is cooled to condense and recover at least a part of distillate. The remaining inert gas mixture is recycled and used for blowing again by means of a blower or the like. In order to perform the operation more effectively, there can be adopted a method in which the remaining uncondensed gas is compressed by a blower or compressor and then cooled to condense and liquefy the majority of the distillate component vapor contained in the gas mixture and recover the fraction of distillate. However, usually vacuum evaporation or vacuum distillation is effected in order to further reduce the boiling point of the distillate, whereby a vacuum pump is used for this purpose instead of the blower or the like. Also in the case where the vacuum pump is employed, compression of the remaining inert gas mixture is effected at its discharge side under atmospheric or elevated pressure, and when this compressed gas is cooled, the majority of the vapor of the distillate contained in the inert gas mixture is condensed and liquefied. In each case, the inert gas mixture containing the distillate vapor saturated at the cooling temperature and its pressure is recycled and used for blowing into the liquid, if necessary after heating.

In the present invention, in the case where a vacuum pump is used in the circulation system of the inert gas, when the inert gas containing a large amount of the solvent fraction is compressed, the fraction is condensed in the vacuum pump. Accordingly, use of a liquid seal type vacuum pump (Nash type) or mechanical vacuum pump is preferred. In

this case, since the distillate is accumulated in the liquid seal tyep pump, the condensed distillate is used as the sealing liquid. The distillate condensed in the pump is recovered in a store tank by overflowing. Accordingly, in the present invention, incorporation of water into a fraction of distillate, which cannot be avoided in case of the steam distillation, can be prevented. In the case where an oil vacuum pump is used, the distillate component is incorporated into oil to reduce the degree of vacuum, and it is necessary to withdraw the used oil to the outside and always supply fresh oil. Therefore, use of this oil vacuum pump is not preferable.

According to the present invention, by performing the evaporation or distillation operation in the inert gas, oxidation or degradation of the components of the liquid by heating can be effectively prevented. Furthermore, since the inert gas is blown into the liquid, the boiling point can be reduced to a level much lower than the boiling point of the intended fraction, and since the evaporation or distillation of the intended fraction can be performed at this low temperature, the degree of oxidation or degradation of the respective components can be controlled to a much lower level, almost nil.

Furthermore, according to the present invention, since an inert gas is used, latent heat for evaporation of water at the evaporation or latent heat for liquefaction of water at the boiling condensation, which is necessary in case of the steam distillation, becomes quite unnecessary, and the evaporation or distillation operation can be accomplished only by giving latent heat for evaporation of the distillate component at the evaporating step and removing this latent heat at the cooling step. Accordingly, the present invention attains various advantages with respect to the heat economy.

The inert gas blown into the liquid not only reduces the boiling point of the distillate component but also act as a carrier gas for transferring this component in the gas phase even if this component is present in a very small amount in the liquid. Accordingly, even a very small amount of the desired distillate component can be evaporated and separated easily. This is another advantage attainable according to the present invention.

The present invention will now be described in detail with reference to the accompanying drawings.

Referring to Figure 1, an apparatus proper 1 comprises a liquid reservoir 2 (tank portion) and a thin film type evaporating zone 3 arranged above the liquid reservoir 2. These zones are provided with liquid-heating steam jackets 4 and 4', respectively. Each of the steam jackets 4 and 4' comprises a steam feed pipe 5 (5'), a valve 6 (6') and a drain withdrawal pipe 7 (7'). By these steam jackets, the respective parts of the apparatus are heated at predetermined temperatures.

Above the thin film type evaporating zone 3, there are laid out a pipe 8 for feeding a liquid to be treated, a valve 9 and a pipe 10. A liquid withdrawal pipe 11 is arranged on the lower end of the liquid reservoir 2, and a liquid circulating route starting from this withdrawal pipe 11, passing through a valve 12, a circulating pump 13, a circulating pipe 14 and a valve 15 and leading to the feed pipe 10 is formed.

Incidentally, in Figure 1, reference numerals 27 and 28 represent gas discharge pipe and valve, respectively, reference numerals 29 and 30 represent inert gas feed pipe and valves, respectively, and reference numeral 31 represents an switch valve.

At first, the valve 15 is closed and the valve 9 is opened to introduce a predetermined amount of a liquid to be treated into the apparatus proper 1 through the pipes 8 and 10. Steam is supplied to the steam jackets 4 and 4' to heat the apparatus proper, and the valve 9 is closed and the valves 12 and 15 are opened. Simultaneously, the pump 13 is actuated and the liquid in the liquid reservoir 2 is circulated to the thin film evaporating zone 3 through the pipes 11, 14 and 10 in the state where heating is performed by the steam jackets 4 and 4'.

Prior to or simultaneously with the above operation, the valve 31 is closed and the valve 30 is opened to blow an inert gas into the liquid through the pipe 29 and boow tube 16 from an inert gas source (not shown) such as a nitrogen bomb. The valve 28 is opened and the liquid seal type vacuum pump 22 is actuated to purge air in the system by the inert gas. Then, the valves 28 and 30 are closed and the valve 31 is opened. The pressure of the blown inert gas is adjusted by the reduction valve 26.

The liquid is stirred by the blown inert gas and a low-boiling-point fraction in the liquid is boiled and evaporated. The inert gas containing the distillate vapor is withdrawn from the apparatus through the pipe 17 and cooled by the condenser 18, whereby the vapor in an amount corresponding to the temperature difference or the vacuum is condensed and is recovered in the first condensed liquid tank 20. The remaining gas is discharged through liquid seal type vacuum pump 22 at atmospheric pressure to condense the majority of the vapor, and the condensed fraction is collected and recovered in the second condensed tank 24. The inert gas left after separation of the condensed liquid is blown into the liquid through the blow tube 16, after the pressure has been reduced to a desired vacuum pressure by the pressure reduction valve 26. Thus, the evaporation is continued.

According to the present invention, by the above-mentioned circulation of the inert gas, separation and recovery of a low-boiling-point fraction of the distillate can be accomplished substantially completely in a short time at a high efficiency.

The present invention will now be described with reference to an example where a liquid mixture comprising a vegetable oil and toluene is treated according to the present invention.

In the evaporation apparatus proper 1 used in this example, the volume of the tank is 100 liters, and the thin film evaporating zone 3 has a diameter of 100 mm and a height of 1000 mm. A liquid mixture comprising 50 kg of a vegetable oil and 40 kg of toluene is charged into this apparatus. The atmosphere of the apparatus is substituted by nitrogen and the vacuum pressure is maintained at 100 Torrs (mmHg), and the temperature of steam supplied to the jackets 4 and 4' is adjusted to 130°C. Under these conditions, the evaporation by blowing nitrogen is

carried out. The temperature of the liquid is 30°C at the start of the operation and 120°C at the termination of the operation. The operation is conducted for 5 hours. As the result, toluene is recovered in an amount of about 40 kg and the toluene content in the vegetable oil as the bottom residue was 500 ppm. The operation can be run continuously, if required.

For comparison, the experiment was carried out by using an apparatus shown in Figure 2 instead of the apparatus shown in Figure 1. The same members as shown in Figure 1 are represented in Figure 2 by the same reference numerals as used in Figure 1. The apparatus shown in Figure 2 is different from the apparatus shown in Figure 1 in the following points. The blow tube 16 shown in Figure 1 is not used, and a steam ejector 32 is used instead of the circulation system shown in Figure 1. The uncondensed gas from the first condensed liquid tank 1 is guided to the ejector 32 through a pipe 33. The ejector 32 is operated by steam fed through a steam feed pipe 34, and the mixture of steam and distillate drain is discharged into a tank 36 through a pipe 35.

The size of the evaporation apparatus used and the composition of the starting liquid are the same as those in the above-mentioned example, and the temperature of steam supplied to the jackets is 130°C. The vacuum pressure is 3 Torrs (mmHg), and the liquid temperature is 30°C at the start of the operation and 120°C at the termination of the operation. The operation was conducted for 10 hours.

The amount recovered of toluene is about 3 kg, and the toluene content in the vegetable oil as the bottom residue is about 5,500 ppm.

The following can be seen from the foregoing results.

Even if an oil containing 44 % of low-boiling-point toluene (having a boiling point of 110°C) is subjected to evaporation under such a high vacuum as 3 Torrs, this low-boiling-point fraction cannot be sufficiently evaporated and separated. Even if the distillation is carried out at an elevated temperature of 120°C under 3 Torrs for 10 hours, about 5,500 ppm of toluene is left in the bottom residue.

Namely, when the content of the low-boiling-point component in the liquid mixture is reduced to a very low level, even if the liquid mixture is heated at the boiling point of this low-boiling-point component or a higher temperature, distillation of this component is hardly accomplished. It is considered that the reason will probably be that a very small amount of the low-boiling-point component is present in the oil in the state adsorbed in the oil.

In contrast, in the case where the evaporation operation is carried out by blowing an inerg gas according to the present invention, even if the vacuum degree is as low as 100 Torrs, the toluene content in the bottom residue is reduced to about 1/10 of the toluene content attained by the customary reduced pressure evaporation and the time required for this evaporation is about 1/2 of the time required at the conventional vacuum evaporation. Moreover, a troublesome operation of separating the desired fraction from water need not be performed and the desired fraction can be recovered in pure form.

The present invention has been described mainly with reference to evaporation. Of course, the present invention can be applied to distillation separation of a liquid containing a plurality of components having boiling points close to each other. In this case, a distillation column such as a bubble cap column, a plate column or a packed column is arranged in the upper portion of the apparatus and the operation is carried out in the same manner as described above.

CLAIMS

1. An evaporation and distillation apparatus for recovering a low-boiling-point fraction of distillate from a liquid containing a plurality of components, said apparatus comprising a heating mechanism to heat the liquid, a blowing mechanism for blowing an inert gas into the liquid and evaporating a low-boiling-point fraction together with the inert gas, an inert gas withdrawal mechanism arranged in the upper portion of the apparatus to withdraw the inert gas containing the low-boiling-point fraction, an inert gas circulating mechanism arranged to connect the inert gas withdrawal mechanism with the blowing mechanism, and a low-boiling-point fraction recovery mechanism arranged in the circulating mechanism to cool and condense the low-boiling-point fraction.

2. Evaporation and distilling apparatus constructed and arranged to operate substantially as herein described with reference to and as illustrated in the accompanying drawings.

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